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February 24, 2005

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APPLICATION NUMBER: 60/545,118

FILING DATE: *February 17, 2004*

RELATED PCT APPLICATION NUMBER: *PCT/US05/03052*



Certified by

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Patent and Trademark Office

Attorney's Docket No. JJA-0308

"PATENT"

Family Number P2004J004

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor's application of: Michael Siskin et al

For: SYNTHESIS OF STERICALLY HINDERED SECONDARY AMINOETHER ALCOHOLS

Commissioner for Patents
Mail Stop: Provisional Patent Application
P.O. Box 1450
Alexandria, Virginia 22313-1450

COVER SHEET FOR FILING PROVISIONAL APPLICATION (37 C.F.R. § 1.53(c))

CERTIFICATION UNDER 37 C.F.R. 1.10 *

(Express Mail label number is mandatory) (Express Mail certification is optional)

<input checked="" type="checkbox"/>	"Express Mail" mailing label number <u>EU863/00789 US</u>	Date of Deposit	<u>February 17, 2004</u>
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- The following comprises the information required by 37 C.F.R. § 1.51(c)(1):
- The name(s) of the inventor(s) is/are:

1. <u>Michael</u>	<u>- -</u>	<u>SISKIN</u>
GIVEN NAME	MIDDLE INITIAL OR NAME	FAMILY (OR LAST) NAME
2. <u>Alan</u>	<u>Roy</u>	<u>KATRITZKY</u>
GIVEN NAME	MIDDLE INITIAL OR NAME	FAMILY (OR LAST) NAME
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- The title of the invention is:

SYNTHESIS OF STERICALLY HINDERED SECONDARY AMINOETHER ALCOHOLS



27810
PATENT TRADEMARK OFFICE

Attorney's Docket No. JJA-0308

Family Number: P2004J004

5. The name, registration, customer and telephone numbers of the attorney is:

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Customer Number 27810

Deposit Account No. 05-1330

6. The docket number used to identify this application is: Docket No. JJA-0308

7. Statement as to whether invention was made by an agency of the U.S. Government or under contract with an agency of the U.S. Government;

This invention was made by an agency of the United States Government, or under contract with an agency of the United States Government.

☒ No.

☐ Yes.

The name of the U.S. Government agency and the Government contract number are:

8. Identification of documents accompanying this cover sheet:

A. Documents required by 37 C.F.R. §§ 1.51(c)(2)-(3):

☒ Specification Number of pages: 38

☐ Drawings Number of sheets:

B. Additional documents:

☒ Claims Number of claims: 36

☐ Assignment

9. Fee


The filing fee for this provisional application, as set in 37 C.F.R. § 1.16(k), is \$ 160.00 for other than a small entity.

10. Method of fee payment

☒ Charge Deposit Account No. 05-1330 in the amount of \$ 160.00.

A duplicate of this Cover Sheet is attached.

Date: 16 Feb 2004


Signature of Attorney

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2/12/2004

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- a -

APPLICATION FOR UNITED STATES PATENT

SYNTHESIS OF STERICALLY
HINDERED SECONDARY AMINOETHER ALCOHOLS

Applicants: Michael Siskin
Alan R. Katritzky
Kostyantyn M. Kirichenko

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Date of Deposit February 17, 2004

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KATHLEEN A. KUNA

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Kathleen A. Kuna

(Signature of person mailing paper or fee)

CASE NO. JJA-0308



27810

PATENT TRADEMARK OFFICE

SYNTHESIS OF STERICALLY
HINDERED SECONDARY AMINOETHER ALCOHOLS

5 FIELD OF THE INVENTION

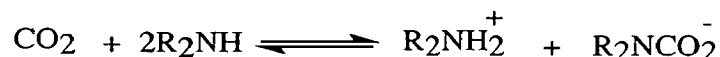
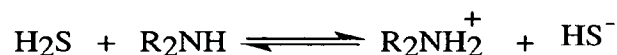
[0001] The present invention relates to a method for the preparation of severely sterically hindered secondary aminoether alcohols which are useful in the removal of hydrogen sulfide from gaseous streams containing hydrogen
10 sulfide and which may also contain carbon dioxide.

DESCRIPTION OF RELATED ART

[0002] It is well-known in the art to treat gases and liquids, such as mixtures
15 containing acidic gases including CO₂, H₂S, CS₂, HCN, COS and oxygen and sulfur derivatives of C₁ to C₄ hydrocarbons with amine solutions to remove these acidic gases. The amine usually contacts the acidic gases and the liquids as an aqueous solution containing the amine in an absorber tower with the aqueous amine solution contacting the acidic fluid countercurrently. Usually
20 this contacting results in the simultaneous removal of substantial amounts of both the CO₂ and H₂S. USP 4,112,052, for example, utilizes a sterically hindered amine to obtain nearly complete removal of CO₂ and H₂S acid gases. This process is particularly suitable for systems in which the partial pressures of the CO₂ and related gases are low. For systems where the partial pressure of
25 CO₂ is high or where there are many acid gases present, e.g., H₂S, COS, CH₃SH, CS₂, etc., a process utilizing an amine in combination with a physical absorbent, referred to as a "non-aqueous solvent process" is practiced. Such a system is described in USP 4,112,051.

[0003] Selective removal of H₂S from acid gas systems containing both H₂S and CO₂, however, is very desirable. Such selective removal results in a relatively high H₂S/CO₂ ratio in the separated acid gas which facilitates the subsequent conversion of the H₂S to elemental sulfur in the Claus process.

5 **[0004]** The typical reactions of aqueous secondary and tertiary amines with CO₂ and H₂S can be represented as follows:



10 where R is the same or different organic radical and may be substituted with a hydroxyl group. Because the reactions are reversible they are sensitive to the CO₂ and H₂S partial pressures which is determinative of the degree to which the reactions occur.

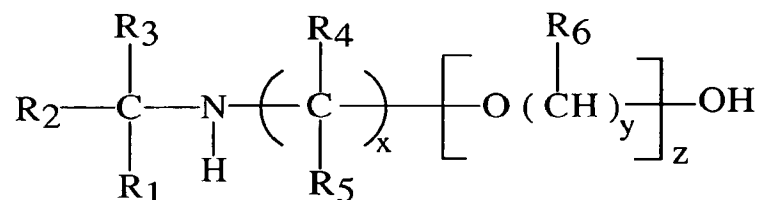
20 **[0005]** Selective H₂S removal is particularly desirable in systems having low H₂S/CO₂ ratios and relatively low H₂S partial pressures as compared to that of the CO₂. The ability of amine to selectivity remove H₂S in such systems is very low.

25 **[0006]** Solutions of primary and secondary amines such as monoethanolamine (MEA), diethanolamine (DEA), diisopropanolamine (DPA), and hydroxyethoxyethylamine (DEA) absorb both H₂S and CO₂, and thus have

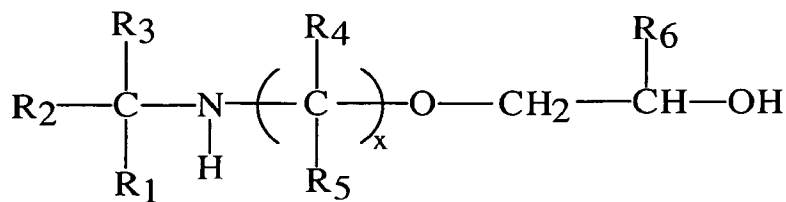
proven unsatisfactory for the selective removal of H₂S to the exclusion of CO₂. The CO₂ forms carbamates with such amines relatively easily.

- 5 [0007] H₂S has been selectively removed from gases containing H₂S and CO₂ by use of diisopropanolamine (DIPA) either alone or mixed with a non-aqueous physical solvent such as sulfolane. Contact times, however, must be kept short to take advantage of the faster reaction of H₂S with the amine as compared to the rate of CO₂ reaction with the amine.
- 10 [0008] Frazier and Kohl, Ind. and Eng. Chem., 42, 2288 (1950) showed that the tertiary amine methyldiethanolamine (MDEA) is more selective toward H₂S absorption as compared to CO₂. CO₂ reacts relatively slowly with tertiary amines as compared to the rapid reaction of the tertiary amine with H₂S. However, it has the disadvantage of having a relatively low H₂S loading capacity and limited ability to reduce the H₂S content to the desired level at low H₂S pressures encountered in certain gases.
- 15 [0009] UK Patent Publication No. 2,017,524A discloses the use of aqueous solutions of dialkylmonoalkanolamines, e.g., diethylmonoethanol amine (DEAE), for the selective removal of H₂S, such material having higher selectivity and capacity for H₂S removal at higher loading levels than MDEA. DEAE, however, has the disadvantage of a low boiling point of 161°C, making it relatively highly volatile resulting in large material loss.
- 20 [0010] USP 4,471,138 the entire teaching of which is incorporated herein by reference, teaches severely sterically hindered acyclic secondary aminoether alcohols having a high selectivity for H₂S compared to CO₂. Selectivity is maintained at high H₂S and CO₂ loadings.

[0011] The severely sterically hindered acyclic amine ether alcohols of USP 4,471,138 are represented by the general formula:



- 5 wherein R₁ and R₂ are each independently selected from the group consisting of alkyl and hydroxyalkyl radicals having 1-4 carbon atoms, R₃, R₄, R₅ and R₆ are each independently selected from the group consisting of hydrogen, alkyl, and hydroxyalkyl radicals having 1-4 carbon atoms, with the proviso that at least one of R₄ or R₅ bonded to the carbon atom which is directly bonded to the nitrogen
- 10 atom is an alkyl or hydroxyalkyl radical when R₃ is hydrogen, x and y are each positive integers ranging from 2-4, and z is a positive integer ranging from 1-4. These materials are prepared by a high temperature reaction preferably in the presence of a solvent, of a secondary or tertiary alkyl primary amine with an ether alcohol containing a carbonyl functionality in the presence of a source of
- 15 hydrogen or with a haloalkoxyalkanol. Preferably the composition is of the general formula:



wherein:

$R_1 = R_2 = R_3 = \text{CH}_3-$; $R_4 = R_5 = R_6 = \text{H}$;

$R_1 = R_2 = R_3 = \text{CH}_3-$; $R_4 = \text{H}$ or CH_3 ; $R_5 = R_6 = \text{H}$;

5 $R_1 = R_2 = R_3 = R_6 = \text{CH}_3-$; $R_4 = R_5 = \text{H}$;

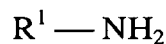
$R_1 = R_2 = R_3 = \text{CH}_3\text{CH}_2-$; $R_4 = R_5 = R_6 = \text{H}$; or

$R_1 \neq R_2 \neq R_3 = \text{H}, \text{CH}_3-, \text{CH}_3\text{CH}_2-$; $R_4 \neq R_5 \neq R_6 = \text{H}, \text{CH}_3-$;

and where $x = 2$ or 3 .

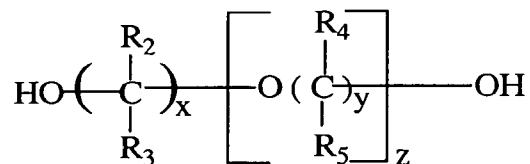
10 **[0012]** U.S. Patent 4,487,967 is directed to a process for preparing severely sterically hindered secondary aminoether alcohols by reacting a primary amino compound with a polyalkenyl ether glycol in the presence of a hydrogenation catalyst at elevated temperatures and pressures. The primary amino compounds employed have a general formula:

15



where R^1 is selected from the group consisting of secondary or tertiary alkyl radicals having 3 to 8 carbon atoms or cycloalkyl radicals having 3 to 8 carbon

20 atoms. The polyalkenyl ether glycols employed have the general formula:



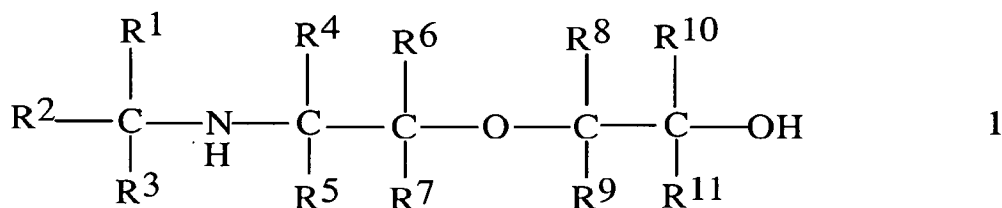
where R_2 , R_3 , R_4 and R_5 are each independently selected from the group consisting of hydrogen, C_1 - C_4 alkyl radicals, and C_3 - C_8 cycloalkyl radicals,

25 with the proviso that if the carbon atom of R_1 directly attached to the nitrogen atom is secondary, at least one of R_2 and R_3 directly bonded to the carbon which is bonded to the hydroxyl group is as alkyl or cycloalkyl radical, x and y are each positive integers independently ranging from 2 to 4 and z is from 1 to 10,

preferably 1 to 6, more preferably 1 to 4. The process is carried out in the presence of a catalytically effective amount of a supported Group VIII metal containing hydrogenation catalyst at elevated temperatures and pressure and the mole ratio of amino compound to polyalkenyl ether glycol is less than 2:1 when
 5 z is greater than 1.

SUMMARY OF THE INVENTION

[0013] A new process is disclosed for the production of severely sterically
 10 hindered secondary aminoether alcohols of the general formula 1:

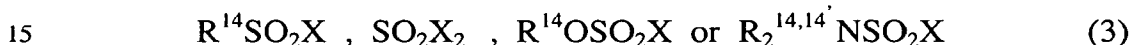


wherein R^1 and R^2 are each independently selected from the group consisting of alkyl and hydroxyalkyl radicals having 1 to 4 carbon atoms, preferably 1 to 2
 15 carbon atoms, or R^1 and R^2 in combination with the carbon atom to which they are attached form a cycloalkyl group having 3 to 8 carbons; R^3 is selected from the group consisting of hydrogen, alkyl or hydroxyalkyl radicals having 1 to 4 carbon atoms, and mixtures thereof, preferably 1 to 2 carbon atoms, preferably alkyl or hydroxyalkyl radicals having 1 to 4 carbon atoms, more preferably 1 to
 20 2 carbon atoms; R^4 , R^5 , R^6 , R^7 , R^8 , R^9 , R^{10} , and R^{11} are the same or different and are selected from hydrogen, alkyl or hydroxyalkyl radicals having 1 to 4 carbon atoms, preferably 1 to 2 carbon atoms, or cycloalkyl radicals having 3 to 8 carbons; R^4 , R^5 , R^6 , R^7 , R^8 , R^9 , R^{10} , and R^{11} are preferably hydrogen provided that when R^3 is hydrogen at least one of R^4 and R^5 bonded to the
 25 carbon directly bonded to the nitrogen atom is an alkyl or hydroxyalkyl radical.

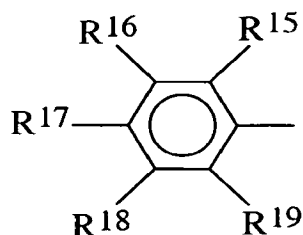
The process involves reacting an organic carboxylic acid or a salt of a carboxylic acid of the formula:



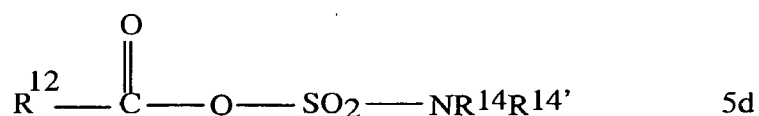
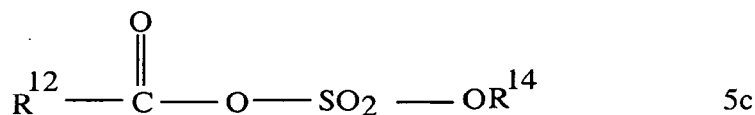
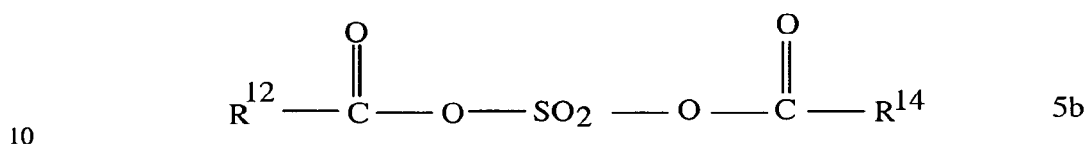
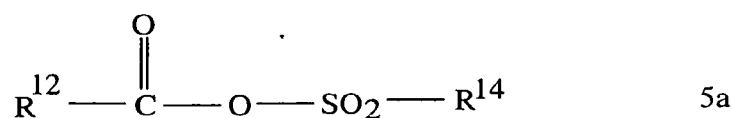
5 wherein R¹² is selected from the group consisting of alkyl radicals having 1 to 4 carbon atoms, preferably 1 to 2 carbon atoms, most preferably methyl, aryl radicals, substituted aryl radicals, preferably phenyl substituted with a hydrogen or one or more alkyl radicals having 1-10 carbon atoms, preferably 1-4 carbon atoms, most preferably methyl in the para position, and mixtures thereof, and Y
10 is selected from the group consisting of hydrogen, alkali metal, ammonium, and mixtures thereof, preferably hydrogen or sodium, with a sulfonyl halide, a sulfuryl halide, a mixed sulfuryl ester halide, or a mixed sulfuryl amide halide of the formula:



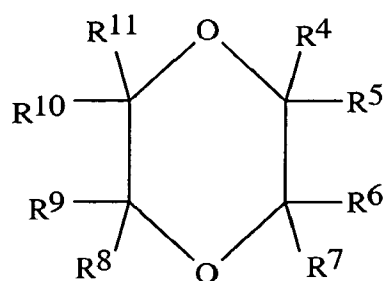
wherein X is selected from the group consisting of F, Cl, Br, I, and mixtures thereof, preferably F, Cl, most preferably Cl, R¹⁴ and R^{14'} are the same or different and each is selected from the group consisting of alkyl radicals having
20 1 to 4 carbon atoms, preferably 1 to 2 carbon atoms, most preferably methyl, haloalkyl radicals of the formula C_nH_{(2n+1)-w}Z_w wherein n is 1 to 4 preferably 1 to 2, and most preferably 1; Z is selected from the group consisting of F, Cl, Br, I, preferably F and Cl, most preferably F; and w ranges from 1 to 5, preferably 1 to 3, most preferably 3, aryl radicals 4



wherein R¹⁵, R¹⁶, R¹⁷, R¹⁸, and R¹⁹ are the same or different and are selected from hydrogen and alkyl radicals having 1 to 20 carbon atoms, preferably R¹⁵, R¹⁶, R¹⁸, and R¹⁹ are hydrogen and R¹⁷ is selected from hydrogen and an alkyl radicals having 1-4 carbons, preferably 1 to 2 carbons, more preferably methyl, and mixtures thereof, to yield sulfonic-carboxylic anhydride compounds of the formula 5:



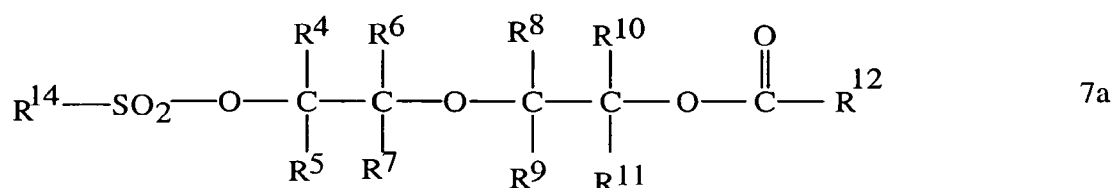
which is then reacted with a dioxane of the formula 6:



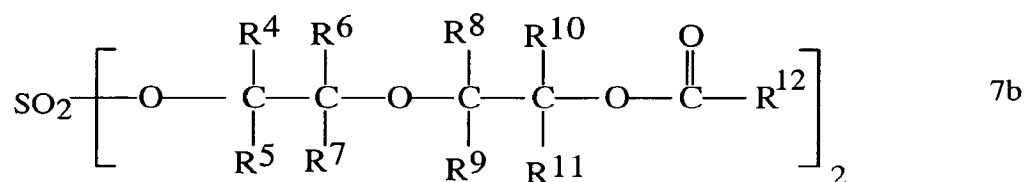
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wherein R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are the same or different and are selected from hydrogen, alkyl and hydroxyalkyl radicals having 1 to 4 carbons, preferably 1 to 2 carbons or cycloalkyl radicals having 3 to 8 carbons, more preferably R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are hydrogen, to yield

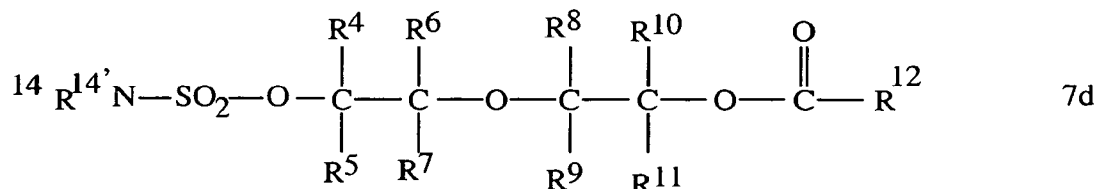
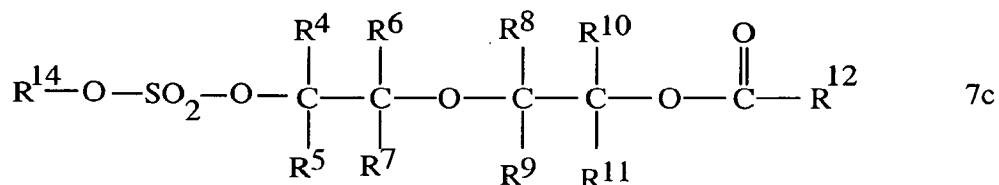
5 cleavage product materials of formula 7



or



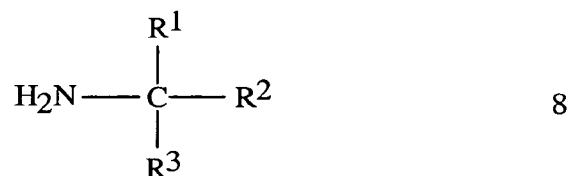
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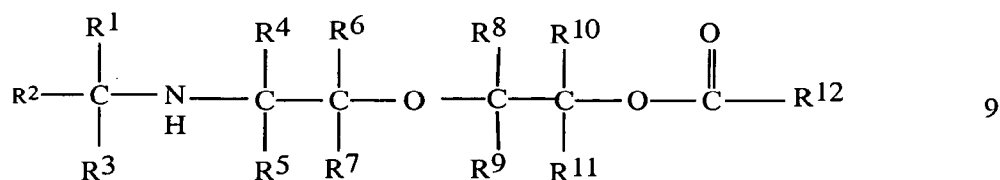
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or mixtures thereof. It is not necessary that the product from each reaction step be isolated before being reacted with the reactant of a subsequent reaction step up to this point. A cleavage product is still produced. The mixing of the organic carboxylic acid or salt thereof with a sulfonyl halide, sulfuryl halide, mixed

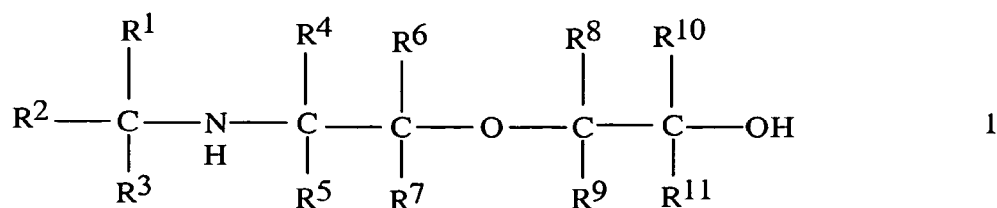
sulfuryl ester halide or mixed sulfuryl amide halide and the dioxane can be in any order or sequence. Thus, the organic carboxylic acid or salt thereof can be mixed with the sulfonyl halide, etc. and then mixed with the dioxane, or the dioxane can be first mixed with the sulfonyl halide, etc. and then the organic carboxylic acid or salt thereof can be added, or the organic carboxylic acid or salt thereof and the dioxane can be mixed followed by the addition of the sulfonyl halide, etc. Thus, the combination of the organic carboxylic acid or salt thereof with the dioxane and the sulfonyl halide, sulfuryl halide, mixed sulfuryl ester halide or mixed sulfuryl amide halide can be combined into a single reaction mixture and reacted as a single mixture in the one step production of the desired cleavage product. This cleavage product is then reacted with an alkylamine of the formula 8:



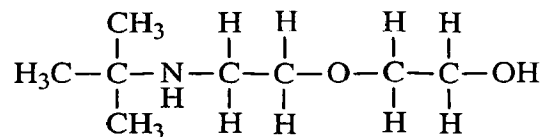
wherein R^1 , R^2 and R^3 are as previously defined to yield 9



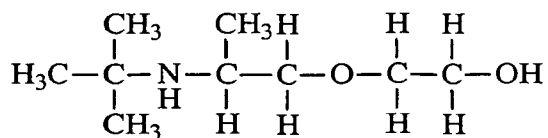
which is subsequently hydrolyzed with a base to yield 1



The preferred compounds defined by the general formula above include:

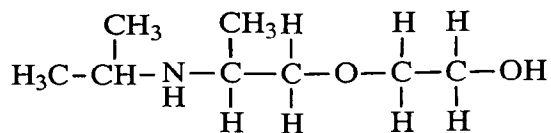


2-(2-*tert*-butylaminoethoxy)ethanol,



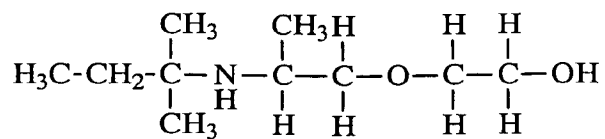
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2-(2-*tert*-butylaminopropoxy)ethanol,

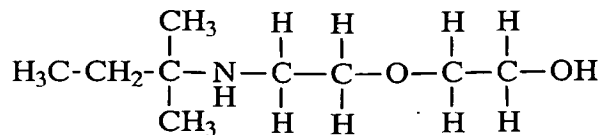


2-(2-isopropylaminopropoxy)ethanol,

10

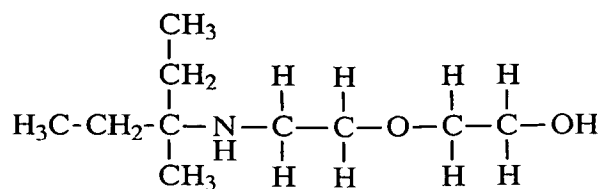


2-[2-(1,1-dimethylpropylamino)propoxy]ethanol,



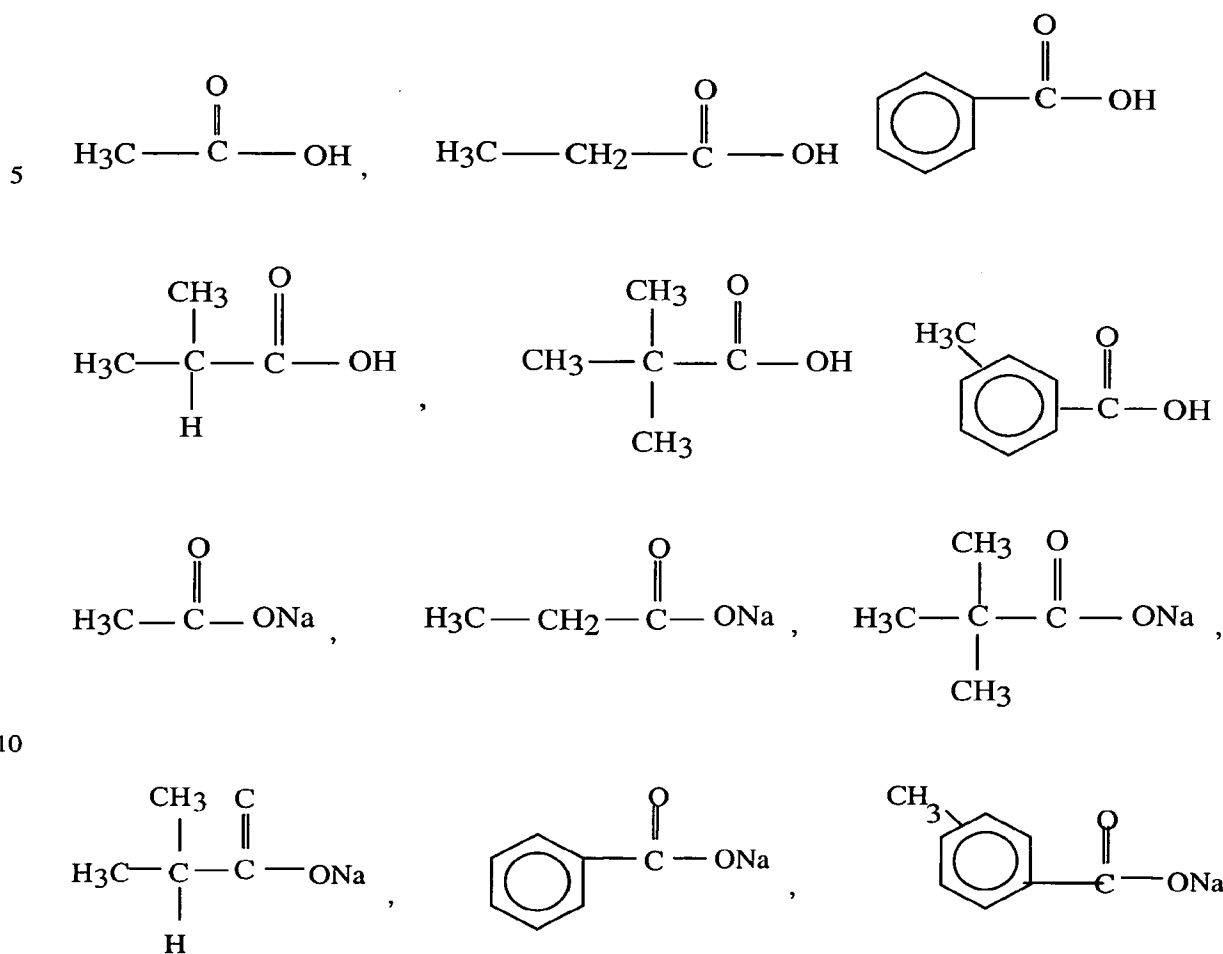
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2-[2-(1,1-dimethylpropylamino)ethoxy]ethanol



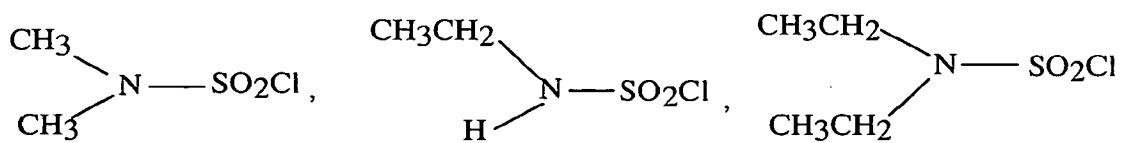
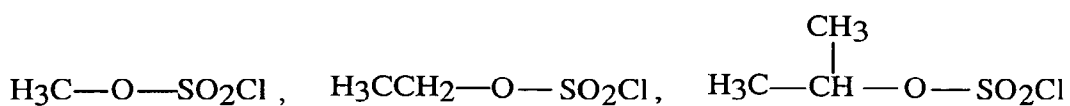
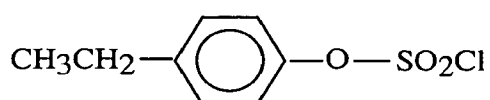
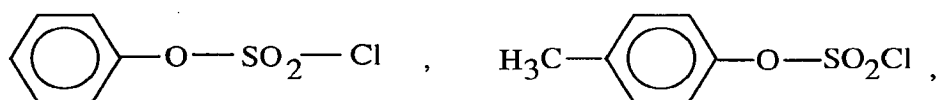
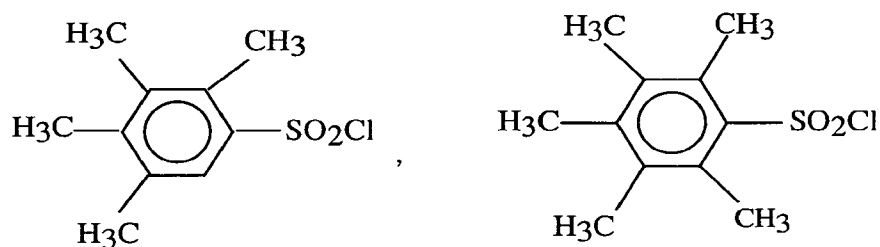
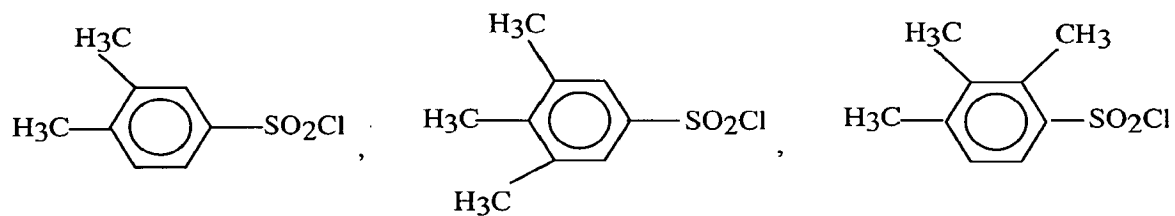
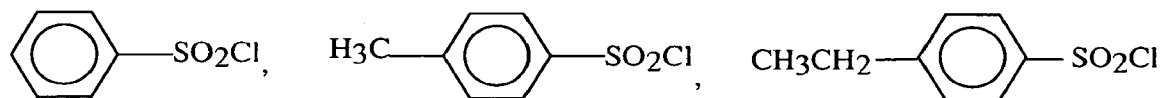
2-[2-(1-ethyl-1-methylpropylamino)ethoxy]ethanol.

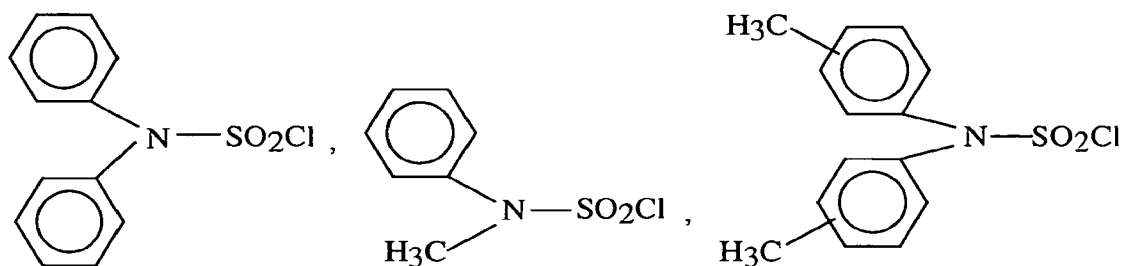
[0014] Typical starting materials to use as the first component are:



[0015] Other materials of the types described above can be readily envisioned.

[0016] This material is then reacted with second component, typically





$\text{H}_3\text{C}-\text{SO}_2\text{Cl}$, $\text{H}_3\text{C}-\text{CH}_2-\text{SO}_2\text{Cl}$, $\text{H}_3\text{C}-\overset{\text{CH}_3}{\underset{|}{\text{CH}}}-\text{SO}_2\text{Cl}$, $\text{F}_3\text{C}-\text{SO}_2$,
 $\text{Cl}_3\text{C}-\text{SO}_2\text{Cl}$, $\text{FCH}_2-\text{CH}_2\text{SO}_2\text{Cl}$, $\text{H}_3\text{C}-\text{CHF}-\text{SO}_2\text{Cl}$,
 $\text{F}_2\text{HC}-\text{CH}_2-\text{SO}_2-\text{Cl}$, $\text{F}_3\text{C}-\text{CF}_2-\text{SO}_2\text{Cl}$, $\text{H}_3\text{C}-\text{CF}_2-\text{SO}_2\text{Cl}$,
 SO_2Cl_2 , SO_2F_2 , SO_2I_2 , SO_2Br_2 and mixtures thereof.

[0017] Similarly, other materials of the type described above can be readily envisioned.

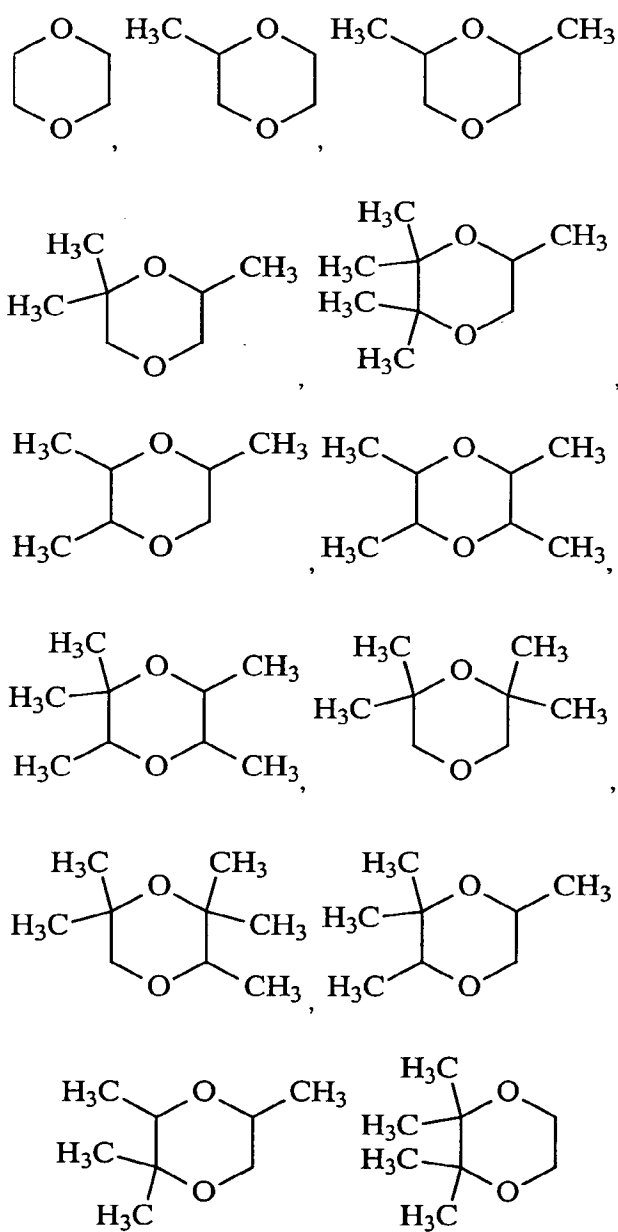
[0018] The reaction of two such components yields acyl sulfonates 5a, 5b, 5c and 5d. The reaction can be conducted at a temperature in the range of between about -20 to 200°C, preferably about -20 to 150°C, more preferably about 0 to 120°C, and a pressure between about 1 bar to 100 bars, preferably about 1 bar to 50 bars, more preferably about 1 bar to 10 bars.

[0019] The reaction can be carried out neat, that is, in the absence of any solvent provided one or the other of the reactants is a liquid and capable of dissolving the non-liquid reactant, or both are liquid. Alternatively, an inert added solvent can be used such as sulfolane, hexanes or acetonitrile. Preferably the dioxane for the subsequent cleavage reaction is used as the solvent resulting in all three reactants being present in a unified first step wherein the reaction mixture contains the dioxane, organic carboxylic acid or salt thereof and the

sulfonyl halide, sulfonyl halide, mixed sulfonyl ester halide or mixed sulfonyl amide halide. This reaction mixture can then be reacted under the conditions subsequently described for the dioxane cleavage reaction, yielding the cleavage product described by general formula 4.

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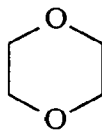
[0020] The sulfonate **5a** and/or **5b** are reacted with a 1,4-dioxane, which is typically of the formula:



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Other substituted isomers can be readily envisioned. Preferably, the 1,4-dioxane is



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[0021] Reaction at elevated temperature is for a time sufficient to cleave the dioxane ring and to achieve about 60-90% conversion to cleavage product. The dioxane also serves as the solvent for the reaction. The molar ratio of dioxane to sulfonate can range from about 1:1 to about 10:1, preferably about 1:1 to about 8:1, most preferably about 1:1 to about 5:1. The reaction can be carried out in the absence of any added solvent, e.g., the dioxane serving as the solvent, or an additional solvent such as acetonitrile or toluene can be used, the reaction being conducted at temperatures between about 50°C to about 200°C, preferably about 70°C to about 160°C, more preferably about 80°C to about 140°C.

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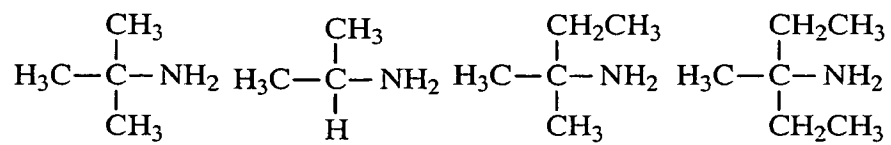
[0022] Preferably, the reaction is carried out in the absence of any added solvent, the dioxane serving as both reactant and solvent, at a temperature in the range of about 50°C to about 160°C, preferably about 70°C to about 160°C, more preferably about 80°C to about 140°C.

20

[0023] The dioxane cleavage step recited above are described in greater detail by Karger and Mazur in "The Cleavage of Ethers by Mixed Sulfonic-Carboxylic Anhydrides", Journal of the American Chemical Society, 1968, 90, 3878-3879. See also, "Mixed sulfonic-carboxylic anhydrides. I. Synthesis and thermal stability. New syntheses of sulfonic anhydrides" Journal of Organic Chemistry, 1971, 36, 528, and "Mixed sulfonic-carboxylic anhydrides. II. Reactions with aliphatic ethers and amines" Journal of Organic Chemistry, 1971, 36, 532.

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[0024] The cleavage product **7a** and/or **7b** and/or **7c** and/or **7d** is then aminated with an amine **8** typically of the formula:



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for a time sufficient to replace the $-\text{O}-\text{SO}_2-\text{R}^{14}$ or the sulfonate group in **7a** and/or **7b** and/or **7c** and/or **7d** by the amine **8**. In the case of the amination of materials **7a**, **7c** and **7d**, the amine to sulfonate group mole ratio is in the range of about 1:1 to about 10:1, preferably about 1:1 to about 8:1, more preferably about 1:1 to 4:1, while in the case of the amination of material **7b** the amine to sulfonate group ratio is in the range of about 2:1 to about 10:1, preferably about 2:1 to about 8:1, more preferably about 2:1 to about 4:1. Expressed differently, in general the amine to group being replaced mole ratio can range from about stoichiometric to about 10:1, preferably about stoichiometric to about 8:1, more preferably about stoichiometric to about 4:1.

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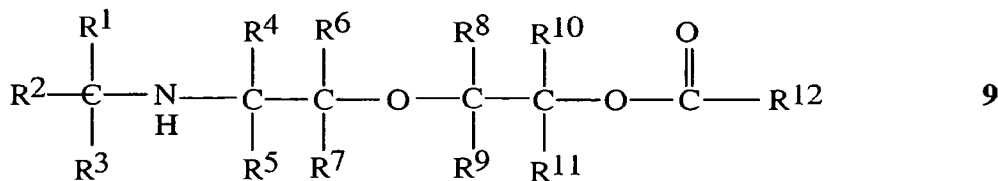
[0025] This amination step can be carried out under any conditions typical in the art. Amination can be conducted at atmospheric or at elevated pressure, elevated pressure being especially suitable when amination is performed using relatively low boiling amines such as *t*-butyl amine.

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[0026] The amination can be conducted at pressures of from about atmospheric (1 bar) to about 100 bars, preferably about 1 to about 50 bars, and at temperatures of from about 40°C to about 200°C, preferably about 40°C to about 125°C. The amination can be performed using reflux, but this is not absolutely necessary. An inert solvent can be optionally used, such as benzene, toluene, diethyl ether, hexane, and the like.

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[0027] Finally, the resultant **9**



is hydrolyzed using a base to yield the final desired product **1**. Typical bases include an alkali metal hydroxide, an alkali metal carbonate, or an alkali metal alkoxide, such as sodium hydroxide, sodium carbonate, sodium methoxide, sodium tert-butoxide, etc. Reaction is conducted at from about 20°C to about 110°C, preferably about 20°C to about 50°C. Reaction under reflux is effective and a desirable technique.

[0028] Use of a solvent is optional for the hydrolysis reaction, one being used if the reactants are not already in the liquid form. Solvents can include water, or alcohol and mixtures thereof.

[0029] If alcohols are used, they can be of the same carbon number or are the same alcohols from which the alkoxide bases themselves are derived. Thus, methanol would be a suitable solvent to use where the base is an alkali methoxide.

EXAMPLES

Cleavage with methyl acetyl sulfate generated in situ using methyl chlorosulfonate and acetic acid in the presence of triethylamine

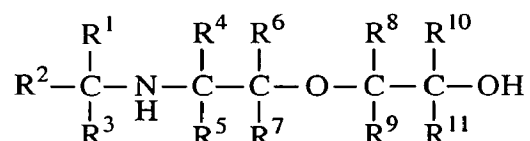
[0030] Methyl chlorosulfonate was prepared according to known procedure [Heller, M. S.; Lorah, D. P.; Cox, C. P., *Chem. Eng. Data*, 1983, 27, 134] by the reaction of sulfuryl chloride with methanol as follow: Methanol (15 g, 0.47 mol) was added dropwise to sulfuryl chloride at 0°C (ice-bath cooling) and the

reaction mixture was stirred at 0°C for 3 hours. After evolution of hydrogen chloride ceased, the reaction mixture was distilled under reduced pressure to give methyl chlorosulfonate (23.5 g, 38 %, bp 62°C / 52–53 mm Hg; lit.[Heller, M. S.; Lorah, D. P.; Cox, C. P., *Chem. Eng. Data*, 1983, 27, 134] 55–56°C / 39 mm Hg). ¹H NMR (CDCl₃) δ 4.21 (s, 3H); ¹³C NMR (CDCl₃) δ 61.2

[0031] Cleavage of dioxane. A 25 mL one-necked flask was charged with acetic acid (0.97 mL, 1.0 g, 16.7 mmol), dioxane (7 mL, 7.0 g, 80 mmol), methyl chlorosulfonate (2.0 g, 15.3 mmol) and then with triethylamine (2.153 mL, 1.56 g, 15.3 mmol) at room temperature under nitrogen. The reaction mixture was refluxed for 72 h. The reaction progress was monitored by NMR. The reaction mixture was then evaporated under vacuum. Dry toluene (15 mL) and *tert*-butylamine (8.4 mL, 5.85 g, 80 mmol) were added to the residue and the mixture was gently refluxed for 24 hours. The reaction mixture was then cooled to room temperature and filtered. The solid was washed with toluene. The combined filtrate was evaporated under vacuum. The residue was extracted with toluene; the extract was filtered and evaporated under vacuum to give 0.3 g of brown oil. The NMR test showed the trace presence of desired product (*t*-BuNHCH₂CH₂OCH₂CH₂OAc). The signals in ¹H NMR spectrum suggest the presence of acetates (HOCH₂CH₂OCH₂CH₂OAc) and/or (AcOCH₂CH₂OCH₂CH₂OAc).

WHAT IS CLAIMED IS:

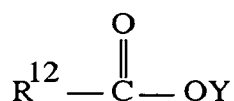
1. A method for the synthesis of severely sterically hindered secondary aminoether alcohols of the formula



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wherein R¹ and R² are each selected from the group consisting of alkyl, hydroxylalkyl radicals having 1 to 4 carbon atoms or in combination with the carbon atom to which they are attached they form a cycloalkyl group having 3 to 8 carbon atoms, and R³ is selected from the group consisting of hydrogen, alkyl hydroxyalkyl radicals having 1 to 4 carbon atoms, and mixtures thereof, and R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰ and R¹¹ are the same or different and are selected from the group consisting of hydrogen, alkyl and hydroxyalkyl radicals having 1 to 4 carbons provided that at least one of R⁴ or R⁵ bonded to the carbon atom directly bonded to the nitrogen atom is an alkyl or hydroxyalkyl radical when R³ is hydrogen, the process involving reacting an organic carboxylic acid or salt of a carboxylic acid of the formula

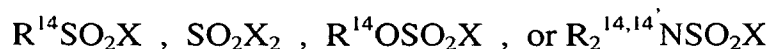
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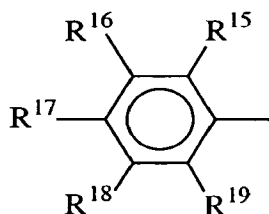
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wherein R¹² is selected from the group consisting of alkyl radicals having 1 to 4 carbon atoms, aryl radicals bearing hydrogen or one or more C₁-C₁₀ alkyl groups substituted thereon, and mixtures thereof, and Y is selected from the group consisting of hydrogen, alkali metal, ammonium, and mixtures thereof, with a sulfonyl halide, a sulfonyl halide, a mixed sulfonyl ester halide, or a mixed sulfonyl amide halide of the formula

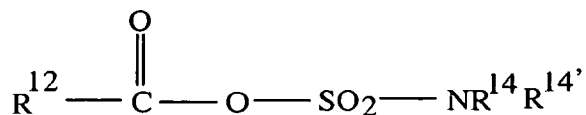
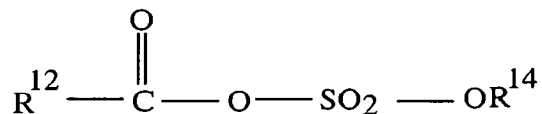
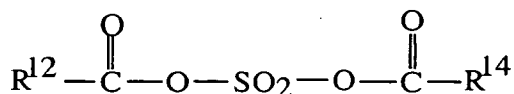
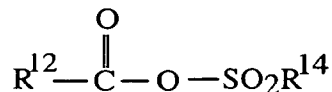
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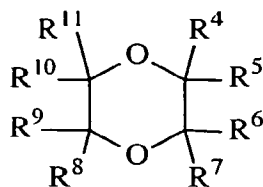
wherein X is selected from the group consisting of F, Cl, Br, I, and mixtures thereof, and R^{14} and $R^{14'}$ are the same or different and each is selected from the group consisting of alkyl radicals having 1 to 4 carbon atoms, haloalkyl radicals of the formula $C_nH_{(2n+1)-w}Z_w$ wherein n is 1 to 4, Z is selected from the group consisting of F, Cl, Br, I, and mixtures thereof, and w ranges from 1 to 5, and aryl radicals



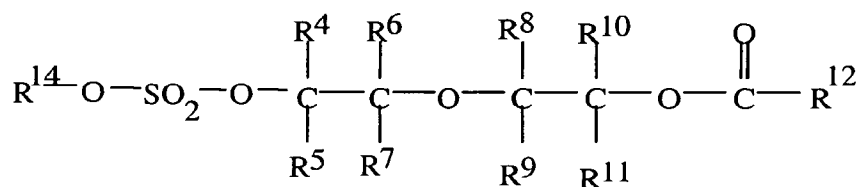
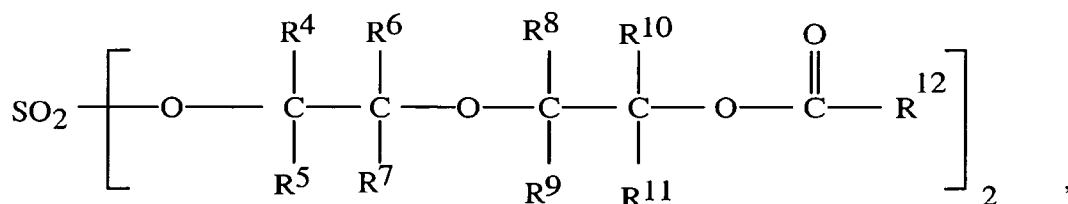
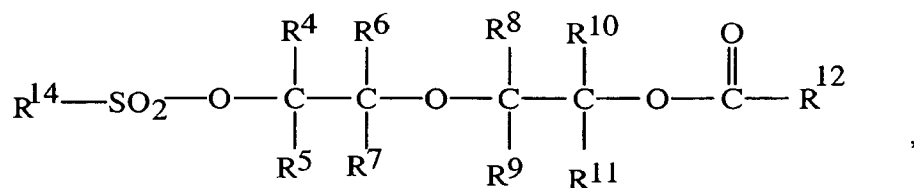
wherein R^{15} , R^{16} , R^{17} , R^{18} , and R^{19} are the same or different and are selected from hydrogen and alkyl radicals having 1 to 20 carbon atoms, and mixtures thereof, to yield material of the general formula



which is then reacted with a dioxane of the formula

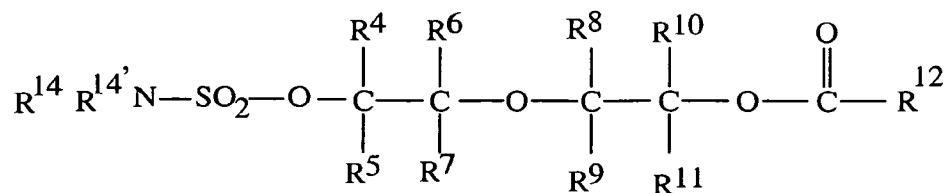


wherein R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are the same or different and are selected from hydrogen, alkyl and hydroxyalkyl radicals having 1 to 4 carbons to
 5 yield

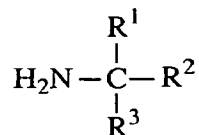


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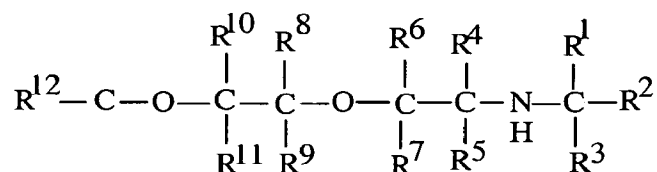
or



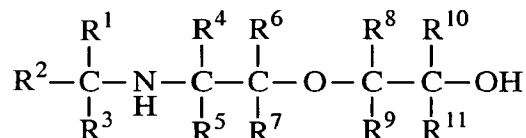
which is then aminated with an alkylamine of the formula



5 wherein R¹, R², and R³ are as previously defined to yield



which is then hydrolyzed with base to yield



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2. The method of claim 1 wherein R¹, R² and R³ are methyl radicals.

3. The method of claim 1 wherein R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are hydrogen.

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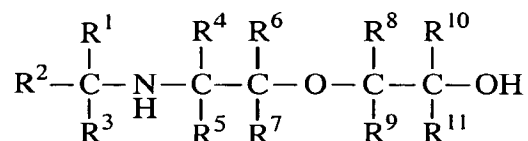
4. The method of claim 1 wherein R¹⁵, R¹⁶, R¹⁸, and R¹⁹ are hydrogen and R¹⁷ is hydrogen or methyl.

5. The method of claim 1 wherein the base is selected from alkali
20 metal hydroxide, alkali metal alkoxide, or alkali metal carbonate.

6. The method of claim 1 wherein Y is hydrogen or sodium.

7. The method of claim 1 wherein R¹, R² and R³ are methyl, R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are hydrogen, R¹⁵, R¹⁶, R¹⁸, and R¹⁹ are hydrogen, R¹⁷ is hydrogen or methyl, and Y is hydrogen, sodium, or ammonium.

5 8. A method for the synthesis of severely sterically hindered secondary aminoether alcohols of the formula



wherein R¹ and R² are each selected from the group consisting of alkyl,
 10 hydroxylalkyl radicals having 1 to 4 carbon atoms or in combination with the carbon atom to which they are attached they form a cycloalkyl group having 3 to 8 carbon atoms, and R³ is selected from the group consisting of hydrogen, alkyl hydroxyalkyl radicals having 1 to 4 carbon atoms, and mixtures thereof, and R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰ and R¹¹ are the same or different and are selected from
 15 the group consisting of hydrogen, alkyl and hydroxyalkyl radicals having 1 to 4 carbons provided that at least one of R⁴ or R⁵ bonded to the carbon atom directly bonded to the nitrogen atom is an alkyl or hydroxyalkyl radical when R³ is hydrogen, the process involving reacting an organic carboxylic acid or salt of a carboxylic acid of the formula

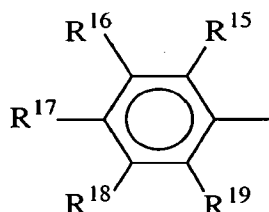


wherein R¹² is selected from the group consisting of alkyl radicals having 1 to 4 carbon atoms, aryl radicals bearing hydrogen or one or more C₁-C₁₀ alkyl groups substituted thereon, and mixtures thereof, and Y is selected from the group

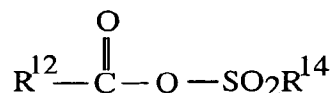
consisting of hydrogen, alkali metal, ammonium, and mixtures thereof, with sulfonyl halide of the formula



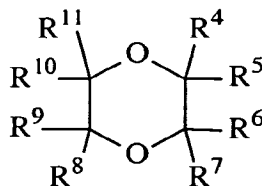
wherein X is selected from the group consisting of F, Cl, Br, I, and mixtures thereof, and R^{14} is selected from the group consisting of alkyl radicals having 1 to 4 carbon atoms, haloalkyl radicals of the formula $C_nH_{(2n+1)-w}Z_w$ wherein n is 1 to 4, Z is selected from the group consisting of F, Cl, Br, I, and mixtures thereof, and w ranges from 1 to 5, and aryl radicals



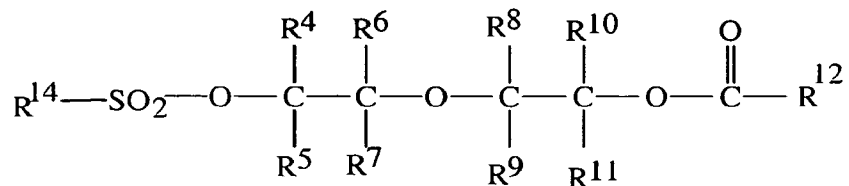
wherein R^{15} , R^{16} , R^{17} , R^{18} , and R^{19} are the same or different and are selected from hydrogen and alkyl radicals having 1 to 20 carbon atoms, and mixtures thereof, to yield material of the general formula



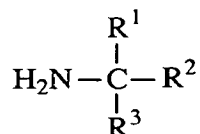
which is then reacted with a dioxane of the formula



wherein R^4 , R^5 , R^6 , R^7 , R^8 , R^9 , R^{10} , and R^{11} are the same or different and are selected from hydrogen, alkyl and hydroxyalkyl radicals having 1 to 4 carbons to yield

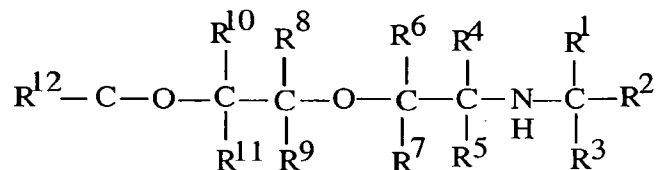


which is then aminated with an alkylamine of the formula

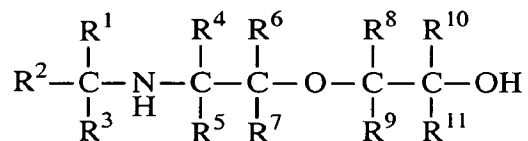


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wherein R^1 , R^2 , and R^3 are as previously defined to yield



which is then hydrolyzed with base to yield



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9. The method of claim 8 wherein R^1 , R^2 and R^3 are methyl radicals.

10. The method of claim 8 wherein R^4 , R^5 , R^6 , R^7 , R^8 , R^9 , R^{10} , and

15 R^{11} are hydrogen.

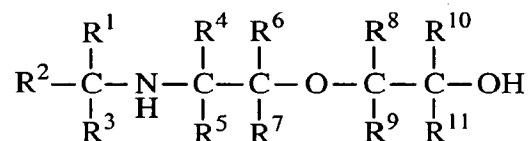
11. The method of claim 8 wherein R^{15} , R^{16} , R^{18} , and R^{19} are hydrogen and R^{17} is hydrogen or methyl.

12. The method of claim 8 wherein the base is selected from alkali metal hydroxide, alkali metal alkoxide, or alkali metal carbonate.

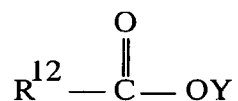
13. The method of claim 8 wherein Y is hydrogen or sodium.

14. The method of claim 8 wherein R¹, R² and R³ are methyl, R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are hydrogen, R¹⁵, R¹⁶, R¹⁸, and R¹⁹ are hydrogen, R¹⁷ is hydrogen or methyl, and Y is hydrogen, sodium, or ammonium.

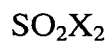
15. A method for the synthesis of severely sterically hindered secondary aminoether alcohols of the formula



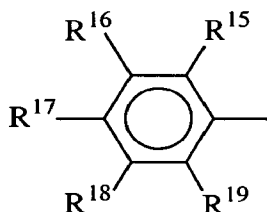
wherein R¹ and R² are each selected from the group consisting of alkyl, hydroxylalkyl radicals having 1 to 4 carbon atoms or in combination with the carbon atom to which they are attached they form a cycloalkyl group having 3 to 8 carbon atoms, and R³ is selected from the group consisting of hydrogen, alkyl hydroxyalkyl radicals having 1 to 4 carbon atoms, and mixtures thereof, and R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰ and R¹¹ are the same or different and are selected from the group consisting of hydrogen, alkyl and hydroxyalkyl radicals having 1 to 4 carbons provided that at least one of R⁴ or R⁵ bonded to the carbon atom directly bonded to the nitrogen atom is an alkyl or hydroxyalkyl radical when R³ is hydrogen, the process involving reacting an organic carboxylic acid or salt of a carboxylic acid of the formula



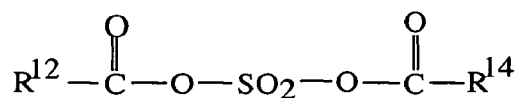
wherein R^{12} is selected from the group consisting of alkyl radicals having 1 to 4 carbon atoms, aryl radicals bearing hydrogen or one or more C_1 - C_{10} alkyl groups substituted thereon, and mixtures thereof, and Y is selected from the group consisting of hydrogen, alkali metal, ammonium, and mixtures thereof, with sulfonyl halide, of the formula



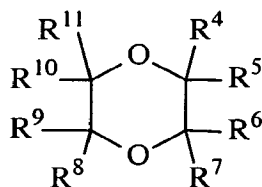
wherein X is selected from the group consisting of F, Cl, Br, I, and mixtures thereof,



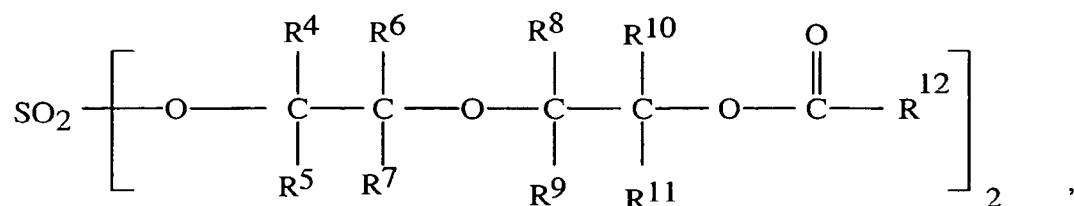
to yield material of the general formula



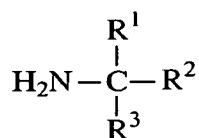
which is then reacted with a dioxane of the formula



wherein R^4 , R^5 , R^6 , R^7 , R^8 , R^9 , R^{10} , and R^{11} are the same or different and are selected from hydrogen, alkyl and hydroxyalkyl radicals having 1 to 4 carbons to yield

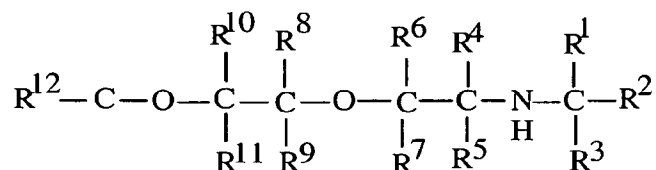


which is then aminated with an alkylamine of the formula

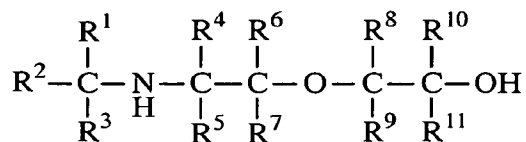


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wherein R¹, R², and R³ are as previously defined to yield



which is then hydrolyzed with base to yield



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16. The method of claim 15 wherein R¹, R² and R³ are methyl radicals.

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17. The method of claim 15 wherein R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are hydrogen.

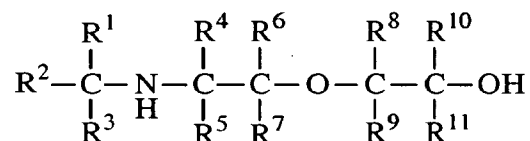
18. The method of claim 15 wherein the base is selected from alkali metal hydroxide, alkali metal alkoxide, or alkali metal carbonate.

19. The method of claim 15 wherein Y is hydrogen or sodium.

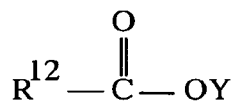
20. The method of claim 15 wherein R¹, R² and R³ are methyl, R⁴,
 5 R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are hydrogen, and Y is hydrogen, sodium, or
 ammonium.

21. A method for the synthesis of severely sterically hindered
 secondary aminoether alcohols of the formula

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wherein R¹ and R² are each selected from the group consisting of alkyl,
 hydroxylalkyl radicals having 1 to 4 carbon atoms or in combination with the
 15 carbon atom to which they are attached they form a cycloalkyl group having 3 to
 8 carbon atoms, and R³ is selected from the group consisting of hydrogen, alkyl
 hydroxyalkyl radicals having 1 to 4 carbon atoms, and mixtures thereof, and R⁴,
 R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰ and R¹¹ are the same or different and are selected from
 the group consisting of hydrogen, alkyl and hydroxyalkyl radicals having 1 to 4
 20 carbons provided that at least one of R⁴ or R⁵ bonded to the carbon atom
 directly bonded to the nitrogen atom is an alkyl or hydroxyalkyl radical when R³
 is hydrogen, the process involving reacting an organic carboxylic acid or salt of
 a carboxylic acid of the formula

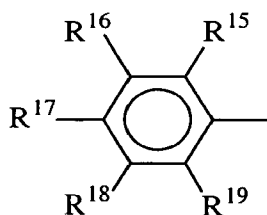


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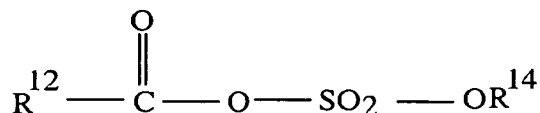
wherein R^{12} is selected from the group consisting of alkyl radicals having 1 to 4 carbon atoms, aryl radicals bearing hydrogen or one or more C_1 - C_{10} alkyl groups substituted thereon, and mixtures thereof, and Y is selected from the group consisting of hydrogen, alkali metal, ammonium, and mixtures thereof, a mixed
 5 sulfuryl ester halide of the formula



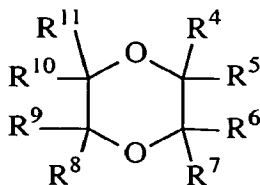
wherein X is selected from the group consisting of F, Cl, Br, I, and mixtures thereof, and R^{14} is selected from the group consisting of alkyl radicals having 1
 10 to 4 carbon atoms, haloalkyl radicals of the formula $C_nH_{(2n+1)-w}Z_w$ wherein n is 1 to 4, Z is selected from the group consisting of F, Cl, Br, I, and mixtures thereof, and w ranges from 1 to 5, and aryl radicals



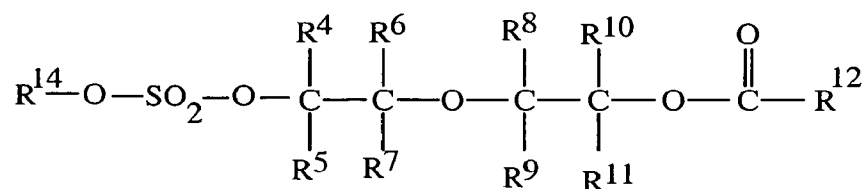
15 wherein R^{15} , R^{16} , R^{17} , R^{18} , and R^{19} are the same or different and are selected from hydrogen and alkyl radicals having 1 to 20 carbon atoms, and mixtures thereof, to yield material of the general formula



20 which is then reacted with a dioxane of the formula

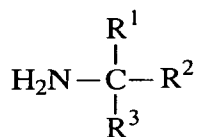


wherein R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are the same or different and are selected from hydrogen, alkyl and hydroxyalkyl radicals having 1 to 4 carbons to yield

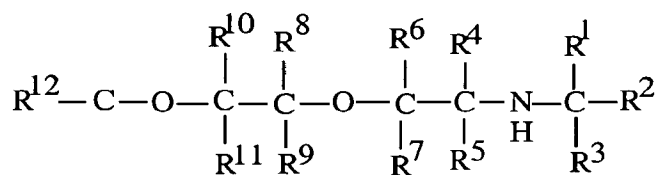


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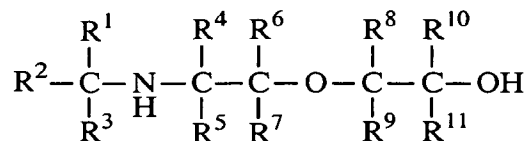
which is then aminated with an alkylamine of the formula



10 wherein R¹, R², and R³ are as previously defined to yield



which is then hydrolyzed with base to yield



15

22. The method of claim 21 wherein R¹, R² and R³ are methyl radicals.

23. The method of claim 21 wherein R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and
20 R¹¹ are hydrogen.

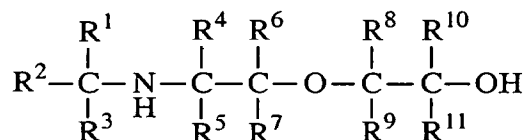
24. The method of claim 21 wherein R¹⁵, R¹⁶, R¹⁸, and R¹⁹ are hydrogen and R¹⁷ is hydrogen or methyl.

5 25. The method of claim 21 wherein the base is selected from alkali metal hydroxide, alkali metal alkoxide, or alkali metal carbonate.

26. The method of claim 21 wherein Y is hydrogen or sodium.

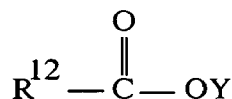
10 27. The method of claim 21 wherein R¹, R² and R³ are methyl, R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are hydrogen, R¹⁵, R¹⁶, R¹⁸, and R¹⁹ are hydrogen, R¹⁷ is hydrogen or methyl, and Y is hydrogen, sodium, or ammonium.

15 28. A method for the synthesis of severely sterically hindered secondary aminoether alcohols of the formula



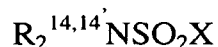
20 wherein R¹ and R² are each selected from the group consisting of alkyl, hydroxylalkyl radicals having 1 to 4 carbon atoms or in combination with the carbon atom to which they are attached they form a cycloalkyl group having 3 to 8 carbon atoms, and R³ is selected from the group consisting of hydrogen, alkyl hydroxyalkyl radicals having 1 to 4 carbon atoms, and mixtures thereof, and R⁴,
25 R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰ and R¹¹ are the same or different and are selected from the group consisting of hydrogen, alkyl and hydroxyalkyl radicals having 1 to 4 carbons provided that at least one of R⁴ or R⁵ bonded to the carbon atom

directly bonded to the nitrogen atom is an alkyl or hydroxyalkyl radical when R³ is hydrogen, the process involving reacting an organic carboxylic acid or salt of a carboxylic acid of the formula

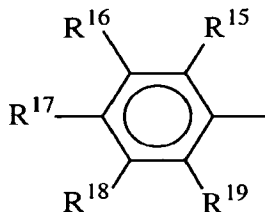


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wherein R¹² is selected from the group consisting of alkyl radicals having 1 to 4 carbon atoms, aryl radicals bearing hydrogen or one or more C₁-C₁₀ alkyl groups substituted thereon, and mixtures thereof, and Y is selected from the group consisting of hydrogen, alkali metal, ammonium, and mixtures thereof, a mixed
10 sulfuryl amide halide of the formula



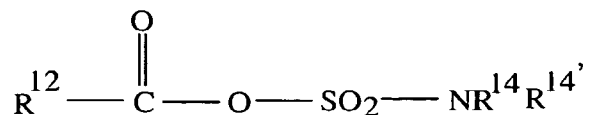
wherein X is selected from the group consisting of F, Cl, Br, I, and mixtures
15 thereof, and R¹⁴ and R^{14'} are the same or different, and each is selected from the group consisting of alkyl radicals having 1 to 4 carbon atoms, haloalkyl radicals of the formula C_nH_{(2n+1)-w}Z_w wherein n is 1 to 4, Z is selected from the group consisting of F, Cl, Br, I, and mixtures thereof, and w ranges from 1 to 5, and
aryl radicals



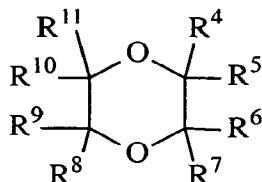
20

wherein R¹⁵, R¹⁶, R¹⁷, R¹⁸, and R¹⁹ are the same or different and are selected from hydrogen and alkyl radicals having 1 to 20 carbon atoms, and mixtures thereof, to yield material of the general formula

25

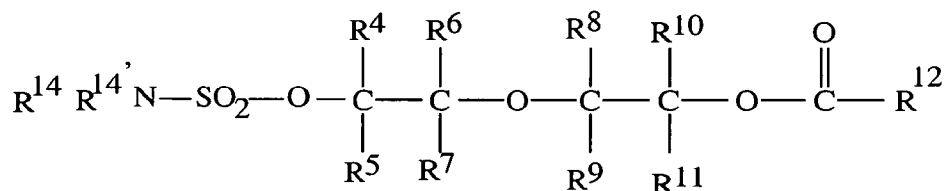


which is then reacted with a dioxane of the formula



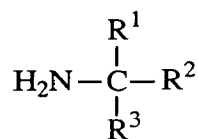
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wherein R^4 , R^5 , R^6 , R^7 , R^8 , R^9 , R^{10} , and R^{11} are the same or different and are selected from hydrogen, alkyl and hydroxyalkyl radicals having 1 to 4 carbons to yield

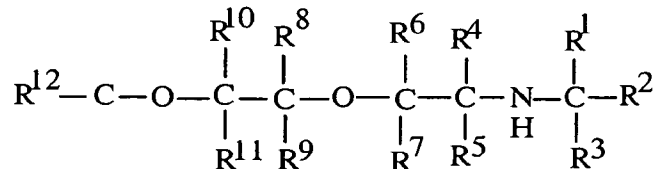


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which is then aminated with an alkylamine of the formula

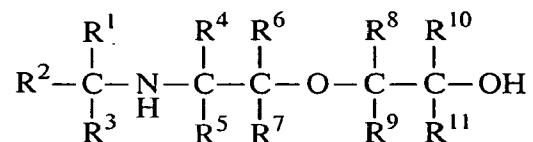


wherein R^1 , R^2 , and R^3 are as previously defined to yield



15

which is then hydrolyzed with base to yield



29. The method of claim 28 wherein R¹, R² and R³ are methyl radicals.

5

30. The method of claim 28 wherein R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are hydrogen.

31. The method of claim 28 wherein R¹⁵, R¹⁶, R¹⁸, and R¹⁹ are hydrogen and R¹⁷ is hydrogen or methyl.

10

32. The method of claim 28 wherein the base is selected from alkali metal hydroxide, alkali metal alkoxide, or alkali metal carbonate.

15

33. The method of claim 28 wherein Y is hydrogen or sodium.

34. The method of claim 28 wherein R¹, R² and R³ are methyl, R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, and R¹¹ are hydrogen, R¹⁵, R¹⁶, R¹⁸, and R¹⁹ are hydrogen, R¹⁷ is hydrogen or methyl, and Y is hydrogen, sodium, or ammonium.

20

35. The method of claims 1, 8, 15, 21 or 28 wherein the acyl sulfonate is made by reacting organic carboxylic acid or the salt of a carboxylic acid with the sulfonyl halide, sulfonyl halide, mixed sulfonyl ester halide or mixed sulfonyl amide halide at a temperature in the range of between about -20 to 200°C at a pressure between about 1 bar and 100 bars, the acyl sulfonate is reacted with the dioxane at a molar ratio of dioxane to acyl sulfonate in the range of 1:1 to 10:1 at a temperature of between about 50°C to about 200°C to yield a cleavage product, the cleavage product and the alkyl amine reacted at an amine to sulfonate group

25

ratio ranging from about stoichiometric to about 10:1 at pressure of from about atmospheric (1 bar) to about 100 bars at temperature of from about 40°C to about 200°C, and the resulting aminated product is hydrolyzed with base at a temperature from about 20°C to about 110°C.

5

36. The method of claim 1, 8, 15, 21 or 28 wherein the organic carboxylic acid or the salt thereof, the sulfonyl halide, sulfonyl halide, mixed sulfonyl ester halide or mixed sulfonyl amide halide and the dioxane are combined in a single step to produce a reaction mixture, the reaction mixture
10 being heated at a temperature of between about 50°C to about 200°C to produce the cleavage product, the cleavage product and the alkylamine are reacted at an amine to cleavage product ratio ranging from about stoichiometric to about 10:1 at a pressure from about atmospheric (1 bar) to about 100 bars at a temperature of from about 40°C to about 200°C, the resulting aminated product being reacted
15 with base at a temperature from about 20°C to about 110°C.

SYNTHESIS OF STERICALLY
HINDERED SECONDARY AMINOETHER ALCOHOLS

5

Abstract of the Disclosure

Severely sterically hindered secondary aminoether alcohols are
10 prepared by reacting an organic carboxylic acid or alkali metal salt of an organic
carboxylic acid with a sulfonyl halide, a sulfuryl halide, a mixed sulfuryl ester
halide or a mixed sulfuryl amide halide to yield a sulfonic-carboxylic anhydride
compound which is then reacted with a dioxane to cleave the ring of the dioxane,
yielding a cleavage product which cleavage product is then aminated with an
15 alkylamine and hydrolyzed with base to yield the severely sterically hindered
secondary aminoether alcohol.